UDC 666.3

# IN VITRO BIOACTIVITY OF CALCIUM PHOSPHATE CERAMICS

# БИОЛОГИЧЕСКАЯ АКТИВНОСТЬ КАЛЬЦИЙ-ФОСФАТНОЙ КЕРАМИКИ IN VITRO

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*Abstract. In this work, the effects of sol-gel derived 52S4.6 bioactive glass on bioactivity of calcium phosphate ceramics was studied. It was found that additive enhance apatite forming ability of ceramics in SBF, active release of calcium cations in PBS, indicating bioresorptivity, does not cause deviation of the pH of PBS. Therefore further research is needed for synthesized ceramics to its in vivo application.*

*Аннотация. В настоящей работе изучено влияние биоактивного стекла 52S4.6, полученного с помощью золь-гель технологии, на биоактивность кальций-фосфатной керамики. Установлено, что добавка усиливает апатитообразующую способность керамики в SBF-растворе, активно высвобождает катионы кальция в PBS-раствор, что свидетельствует о биорезорбтивности, не вызывает изменения pH PBS-раствора. Следовательно, необходимы дальнейшие исследования синтезированной керамики для ее применения in vivo.*

Calcium phosphate ceramics has been widely used for bone substitution because of non-toxicity, excellent bioactivity, resorbability, biocompatibility [1, 2].

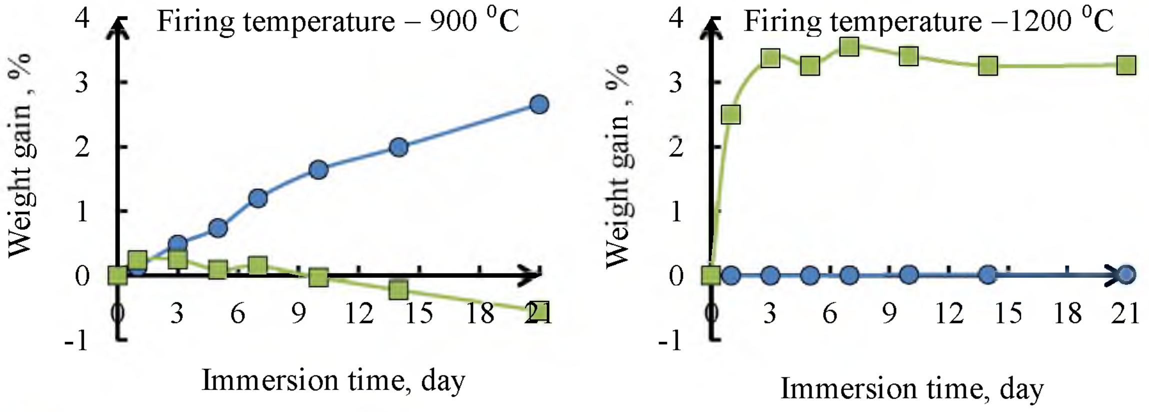
The aim of our work is development of compositions and technological parameters for the production of calcium phosphate porous ceramics with improved mechanical properties using of 3D printing.

To prepare of slurry for 3D printing hydroxyapatite was synthesized by aqueous precipitation. And moreover, sol-gel derived 52S4.6 bioactive glass was used as an additive. Before reaching the gel point, the sol was mixed with hydroxyapatite. The amount of additives was 10.0–30.0 wt.%. The printed samples were dried at 50–70 °C for 24 h and fired at 900–1200 °C for 8 h.

Previous research has shown that sample containing 10 wt.% of the sol-additive had optimal physicochemical properties: water absorption – 6.1–33.1%; apparent porosity – 49.1–14.3 %; bulk density – 1480–2340 kg/m$^3$; compressive strength – 10.2–44.9 MPa. Phase composition of synthesized ceramics – hydroxyapatite and β-tricalcium phosphate.

As it is known, materials for bone substitution should produce apatite on its surface in the living body and bonds to living bone through this apatite layer [3]. The apatite forming ability of composition optimized ceramics was investigated by examining apatite formation on the surface of the samples treated in simulated body fluid (SBF). The samples were weighed, placed in plastic containers containing 100 cm3 of SBF and kept for 1–21 days at 37 $^0$C in the TS-1/20 thermostat.

Figure 1 shows the weight gain of the samples after immersion in SBF solution. The heat treatment temperature and the composition of the samples affect the rate of apatite formation. The apatite layer formed on the surface of ceramics sintered without additive only at a temperature of 900 °C, which may be due to phase structure of obtained material. As the firing temperature increases, the amount of $\beta$-$Ca_3(PO_4)_2$ increases. The experimental results demonstrated that, during soaking in SBF, the specimens obtained using sol-additive at 900 °C dissolved. However, samples containing sol-additive and firing at 1200 °C enhanced the rate of mineralization. This is due to the fact that the apatite forming ability depends on hydroxyapatite / β-tricalcium phosphate ratio. The weight gain of samples after immersion in SBF solution for 21 days was 3.5 %.



● – without additives; 🟩 – sol-additive

Figure 1 – Weight gain of samples after immersion in SBF solution

To study behavior in a phosphate-buffered saline (PBS) (pH = 7.4), weighed samples were placed in specified solution for 1–21 days at 36.6 °C. Complexometric titration and X-ray fluorescence spectrometry (Axios, PANalytical, Netherlands) were used to find the total calcium ion content of PBS solution (figure 2).

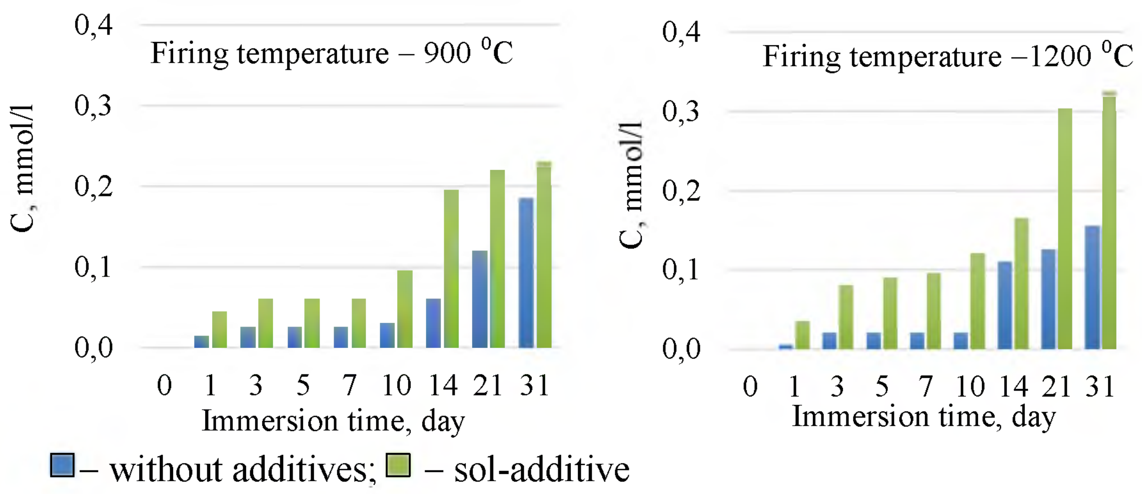


Figure 2 – Concentration of calcium ions in solution after immersion of samples

Ceramic samples with sol-additive showed a pronounced loss of weight and active release of calcium cations, which confirms their ability to bioresorb. The rate of ceramics resorption varied from $0{,}9 \cdot 10^{-4}$ to $3{,}7 \cdot 10^{-4}$ g/day depending on the firing temperature.

A significant deviation of the pH from a neutral value, both towards increased alkalinity and towards acidity, negatively affects the viability of tissues and limits the possibility of using the material in direct contact with them inside the body.

Figure 3 illustrates that ceramics synthesized at 900 °C displayed highly alkaline pH. In contrast, specimens firing at 1200 °C exhibited weakly alkaline medium throughout all the experimental intervals.

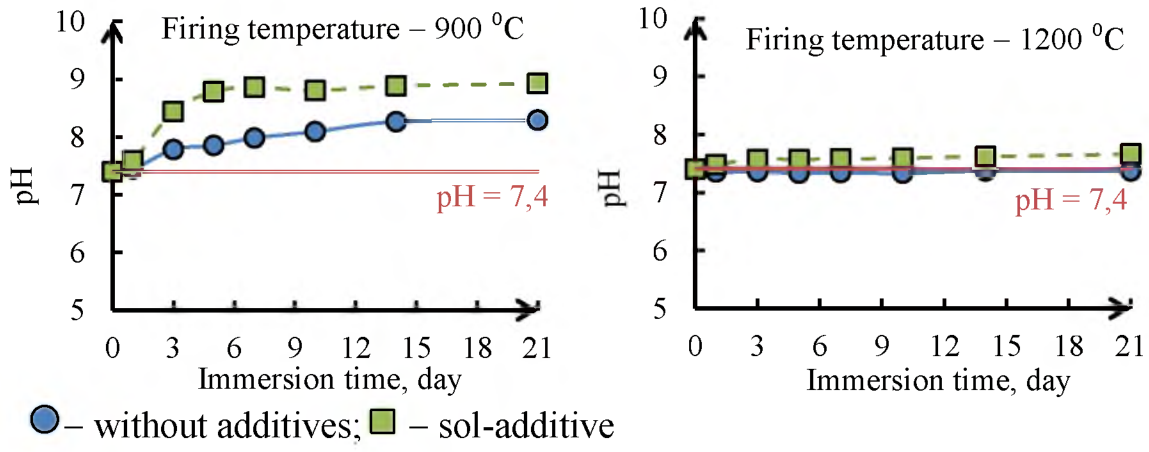


Figure 3 – Change of pH of solution after immersion of samples

Thus, the synthesized calcium phosphate ceramics can be recommended for further medical research in order to study the possibility of their use in bone surgery.

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